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Entry 5 of 16

File: USPT

Mar 19, 1996

DOCUMENT-IDENTIFIER: US 5500062 A

TITLE: Emulsion explosive

**BSPR:**

Suitable chemicals for the in situ generation of gas bubbles include peroxides, such as hydrogen peroxide, nitrites, such as sodium nitrite, nitrosoamines, such as N,N'-dinitrosopentamethylenetetramine, alkali metal borohydrides, such as sodium borohydride, and carbonates, such as sodium carbonate. Preferred chemicals for the in situ generation of gas bubbles are nitrous acid and its salts which decompose under conditions of acid pH to produce nitrogen gas bubbles. Preferred nitrous acid salts include alkali metal nitrites, such as sodium nitrite. These can be incorporated as an aqueous solutions a pre-emulsified aqueous solution in an oil phase, or as a water-in-oil micro emulsion comprising oil and nitrite solution. Catalytic agents such as thiocyanate or thiourea may be used to accelerate the decomposition of a nitrite gassing agent. Suitable small hollow particles include small hollow microspheres of glass or resinous materials, such as phenol-formaldehyde, urea-formaldehyde and copolymers of vinylidene chloride and acrylonitrile. Suitable porous materials include expanded minerals such as perlite, and expanded polymers such as polystyrene.

**CCOR:**149/46**CCXR:**149/60**ORPL:**

"Ullmann's Encyclopedia of Industrial Chemistry", Fifth, Completely Revised Edition, vol. A9: Dithiocarbamic Acid to Ethanol, pp. 306-325.

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Entry 8 of 16

File: USPT

Jul 24, 1990

DOCUMENT-IDENTIFIER: US 4942800 A

TITLE: Process for the manufacture of explosive cartridges, and explosive cartridges obtained using the said process

**ABPR:**

A process for the manufacture of an explosive cartridge in a tubular casing, including mixing a solution containing hydrogen peroxide, an oxidizable organic material, and at least one precursor monomer of a gelling agent; and reacting the at least one precursor monomer to form macromolecules of the gelling agent in situ in the tubular casing.

**BSPR:**

The present invention relates to a process for the manufacture of explosive cartridges containing, in a tubular casing, an explosive composition comprising hydrogen peroxide, an oxidizable organic material and a gelling agent. More particularly, it relates to a process where the gelling agent belongs to the class of macromolecular plastic materials.

**BSPR:**

There have long been known explosive compositions which contain an aqueous solution of concentrated hydrogen peroxide combined with a finely divided oxidizable combustible material and an absorbent filler material (U.S. Pat. No. 3,047,441 to A. W. Baker et al. at column 2, lines 1 to 10 and column 6, claim 1). Among the absorbent filler materials employed, mention is made of the use of gel-forming products (column 2 lines 60 to 72), as well as of thermosetting or thermoplastic synthetic resins such as urea-formaldehyde, phenol-formaldehyde or polymethyl methacrylate resins (column 2, lines 54 to 58). In an alternative form, combustible materials capable of also acting as the filler material may be employed (column 2, lines 26 to 31).

**BSPR:**

To this end, the invention relates to a process for the manufacture of explosive cartridges containing, in a tubular casing, an explosive composition comprising hydrogen peroxide, at least one oxidizable organic material and at least one gelling agent belonging to the class of macromolecular plastic materials; according to the invention, in a first stage, hydrogen peroxide, the oxidizable organic material and at least one precursor monomer of the gelling agent are mixed and, in a second stage, the macromolecules of the gelling agent are synthesized in situ in the tubular casing.

**BSPR:**

Hydrogen peroxide is employed in the process according to the invention in the form of a concentrated solution of hydrogen peroxide in a solvent. "Concentrated solutions" means solutions where the

weight content of hydrogen peroxide is higher than 60%. Solutions containing at least 65% by weight and up to 99.9% by weight of hydrogen peroxide are highly suitable. Solutions which contain between 70 and 90% by weight of hydrogen peroxide are preferably employed. The solvent for hydrogen peroxide may be water or an inert organic solvent. Examples of suitable organic solvents are n-butanol, acetonitrile and chloroform. Water is the preferred solvent because of its negligible cost and of its lesser risks associated with its use in the presence of concentrated solutions of hydrogen peroxide.

BSPR:

In the process according to the invention, the oxidizable organic material employed consists of any organic material capable of oxidizing rapidly in the presence of hydrogen peroxide or else a mixture of two or more of these materials. The materials capable of releasing large volumes of gas after oxidation are highly suitable. In order to restrict atmospheric pollution after the detonation, it is advantageous to choose organic products containing only carbon, hydrogen and oxygen.

BSPR:

An essential characteristic of the process according to the invention is to obtain, at the end of the first stage, a mixture which is as homogeneous as possible before proceeding with the synthesis of the macromolecules of the gelling agent. To this end, it is most particularly preferable to choose the oxidizable organic material among the organic materials belonging to the class of the products which are normally liquid at the temperature at which the first stage is carried out and which are miscible in high proportions with the hydrogen peroxide solution and to the class of the products which are soluble in a high proportion in this solution. Examples of such organic materials are the lower aliphatic alcohols such as methanol and ethanol, aliphatic diols containing fewer than 5 carbon atoms, such as ethylene glycol, and sugars such as pentoses and hexoses, especially sucrose.

BSPR:

In the process according to the invention, the gelling agent is a macromolecular plastic material which is synthesized in situ in the tubular casing, in the presence of hydrogen peroxide and of the oxidizable organic material, from one or more precursor monomers.

BSPR:

To this end, the monomer is incorporated in the mixture in the first stage of the process. Monomers which are miscible and/or soluble in the hydrogen peroxide solution at the temperature at which both stages of the process are performed are advantageously chosen.

BSPR:

According to the invention, it is important that the synthesis of the macromolecules should take place in the mass of the combined mixture introduced into the tubular casing. To this end, it is advantageous to choose the method of synthesis in solution. Where appropriate, the synthesis of the plastic material is carried out in the hydrogen peroxide solution. In this form of embodiment of the process according to the invention, the other constituents of the explosive mixture may be dispersed in the solid or liquid state in the hydrogen peroxide solution. It is preferable that they should be in the dissolved form in the hydrogen peroxide solution. Plastic materials which may be obtained by this method are generally well known as such. They belong to the classes of resins which are usually produced by solution polymerization. A category of resins which are particularly suitable are those obtained by aqueous solution polymerization, with the hydrogen peroxide solution then being an aqueous solution. In this category, water-soluble resins are of very

particular value.

BSPR:

By virtue of its organic nature, the gelling agent itself also constitutes an oxidizable material. In an alternative embodiment of the process according to the invention, the hydrogen peroxide is mixed, in the first stage, with the precursor monomer of the gelling agent, to the exclusion of all other oxidizable organic materials.

BSPR:

In the process according to the invention, a small proportion of additives is incorporated in the mixture, generally in the first stage, this proportion being generally lower than 5% by weight of this mixture. The principal purpose of these additives is to stabilize the hydrogen peroxide against slow decomposition into water and oxygen. Materials which are known for stabilizing concentrated hydrogen peroxide solutions, such as phosphates, stannates and sequestrants for heavy metals, of an organic or inorganic type, are employed for this purpose. Other additives may be added to the mixture to impart to the explosive material manufactured special properties such as a lower friction or impact sensitivity, a reduced tendency to sweat, improved mechanical properties such as plasticity, and resistance to frost and to low temperatures in general.

BSPR:

The proportions of the various constituents to be employed in the mixture depend, at the same time, on the nature of the oxidizable organic material, on that of the precursor monomer of the plastic material forming the gellant, and on the solvent(s) present. They can easily be determined by laboratory formulation tests. As a general rule, the formulation of the mixture must be adapted so that the respective quantities of hydrogen peroxide, on the one hand, and of the materials capable of being oxidized (which include the oxidizable material, the plastic material and any organic solvent present), on the other hand, are not too remote from the stoichiometric quantities corresponding to the chemical reactions of oxidation by hydrogen peroxide.

BSPR:

To this end, the mixture produced in the first stage of the process generally contains a weight proportion of 50 to 95% of a concentrated solution of hydrogen peroxide in water or in an organic solvent, 2 to 40% of oxidizable organic material, 2 to 40% of precursor monomer of the plastic material and 0 to 5% of additives.

BSPR:

The best results have been obtained using mixtures in which the weight proportions comprise 65 to 85% of concentrated solution of hydrogen peroxide, 5 to 30% (preferably 5 to 20%) of oxidizable organic material, 5 to 30% (preferably 5 to 20%) of precursor monomer of the plastic material and not more than 1% (preferably 0.2%) of additives, such as defined above.

BSPR:

In a particular embodiment of the process according to the invention, which is preferred, the oxidizable organic material, the solvent, the precursor monomer of the plastic material and the respective proportions of these constituents and of hydrogen peroxide are chosen so that the mixture obtained in the first stage comprises only a single homogeneous liquid phase with low dynamic viscosity, for example below 1,500 Pa.s, preferably below 1,000 Pa.s.

BSPR:

The order in which the various constituents are introduced into the mixture must be adapted to the nature of the constituents and to type

of mixer. In most cases, the oxidizable organic material is first mixed with the stabilizing additive where appropriate. Hydrogen peroxide is then added progressively into the mixer, followed by the precursor monomer of the plastic material.

BSPR:

The order of addition of hydrogen peroxide and of the monomer may also be reversed so as to end the first stage of the process according to the invention by introducing the hydrogen peroxide solution.

DEPR:

293.8 g of ethylene glycol and 1.3 g of diethylenetriaminepenta(methylenephosphonic) acid (Briquest 543-45 AS, registered trademark of Albright & Wilson) were introduced into a stainless steel planetary mixer of registered trademark Hobart, model N-50. After the mixing of the constituents was started, 993.2 g of an aqueous solution of hydrogen peroxide containing 85% by weight of H.sub.2 O.sub.2 was then introduced in the form of a slow and continuous stream, followed by 13 g of polyacrylic acid (Carbopol 934, registered trademark of B.F. Goodrich Cy). The introduction of all the components of the mixture took approximately 15 min.

DEPR:

262.6 g of methanol and 1.3 g of diethylenetriaminepenta(methylenephosphonic) acid (Briquest 543-45 AS) were introduced with stirring into a 1,800 ml beaker equipped with a rotary stirrer consisting of a glass paddle. After a few minutes, 1,024.4 g of an aqueous solution of hydrogen peroxide containing 85% by weight of H.sub.2 O.sub.2 was introduced in a slow and continuous stream. 3.80 g of crosslinked polyacrylamide (Aquasorb PR 3005, registered trademark of SNF Floerger) were then placed in the bottom of the tubular casings.

DEPR:

After 376.2 g of the liquid mixture of methanol and of stabilized hydrogen peroxide were transferred by pouring into each casing, the contents of the casings were carefully mixed, the cartridges were closed and were stored for 24 hours at ambient temperature, during which the gelling of the mixture was completed. The same test to measure the detonation velocity as in Example 1R was then carried out.

DEPR:

A mixture of 327.6 g of sucrose, 0.86 g of diethylenetriaminepenta(methylenephosphonic) acid (Briquest 543-45 AS), and 959.4 g of an aqueous solution of hydrogen peroxide containing 85% by weight of H.sub.2 O.sub.2 was prepared according to the same process as that of Example 2R. 3.8 g of crosslinked polyacrylamide (Aquasorb PR 3005) was then introduced into the bottom of the tubular casings.

DEPR:

After 376.2 g of the liquid mixture of sucrose in solution in the stabilized hydrogen peroxide were transferred by pouring into each casing, the contents of the casings were carefully mixed, the cartridges were closed and were stored for 24 hours at ambient temperature. The cartridges were then subjected to the same detonation velocity measurement test as in Examples 1R and 2R.

DEPR:

195 g of sucrose and 0.39 g of dipicolinic acid were introduced into a 1,800 ml beaker. After stirring for a few minutes, 975 g of an aqueous solution of hydrogen peroxide containing 85% by weight of H.sub.2 O.sub.2 and 130 g of acrylic acid monomer were then

introduced successively into the beaker, in the form of a slow and continuous stream.

## DEPR:

After stirring for a few minutes, 924 g of an aqueous solution of hydrogen peroxide containing 85% by weight of H.sub.2 O.sub.2 were introduced into the beaker, in the form of a slow and continuous stream, followed successively by 72 g of acrylic acid monomer and 18 g of tripropylene glycol diacrylate (TPGDA).

## CLPR:

1. Process for the manufacture of explosive cartridges containing, in a tubular casing, an explosive composition comprising hydrogen peroxide, at least one oxidizable organic material and at least one gelling agent belonging to the class of macromolecular plastic materials, comprising the steps of:

## CLPR:

10. Process according to claim 1 wherein a stabilizing agent for hydrogen peroxide is incorporated in the mixing step.

## CLPR:

11. Process according to claim 10, wherein in the mixing step, the resulting mixture comprises from 65 to 85% by weight of an aqueous solution comprising about 85% by weight of hydrogen peroxide; 5 to 20% by weight of sucrose; 5 to 20% by weight of acrylic acid; and 0.01 to 0.2% by weight of dipicolinic acid.

## CLPV:

mixing (a) a solution containing hydrogen peroxide of at least about 60 weight %, (b) oxidizable organic material and (c) at least one precursor monomer of the gelling agent; and

## CCXR:

149/109.6

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## Document Number 1

Entry 1 of 11

File: USPT

Mar 17, 1998

DOCUMENT-IDENTIFIER: US 5727368 A

TITLE: Hybrid motor system with a consumable catalytic bed a composition of the catalytic bed and a method of using

### ABPL:

A method, device and composition for achieving ignition and sustained combustion using a solid consumable catalytic bed (CCB) is described. The CCB accomplishes this by decomposition of an injected fluid. Initially this reaction is catalytic or hypergolic in nature, eventually becoming entirely thermal as the CCB is consumed in the reaction generated. The CCB may be placed in a hybrid motor system such that the decomposed injected fluid will undergo combustion with the solid grain. When the injected fluid is high concentration hydrogen peroxide the catalytic materials of construction for the CCB are selected from the metallic hydride family, the manganese oxide family and the cuprocyanide family.

### BSPR:

The invention described herein relates to a hybrid motor system that uses either a fluid oxidizer and a solid fuel or a solid oxidizer and fluid fuel. In one embodiment, the fluid is an oxidizer consisting of high concentration hydrogen peroxide. Hydrogen peroxide has the unique property that it will undergo decomposition into oxygen, water and energy in the presence of certain chemical catalysts. If the concentration of hydrogen peroxide is high enough, this decomposition will produce hot gas sufficient to spontaneously initiate combustion of a fuel. Commercially available hydrogen peroxide typically includes materials such as stannates and phosphates in order to reduce the reactivity of the hydrogen peroxide in the presence of contaminants. These materials are often referred to as 'stabilizers'. Commercial grade or stabilized hydrogen peroxide contains approximately 5 to 100 ppm of stabilizers. United States military specification grade hydrogen peroxide contains less than 5 ppm of any stabilizers and is commonly referred to as 'unstabilized'. The concept of this invention is not limited to the use of either stabilized or unstabilized hydrogen peroxide. Other oxidizer and fuel combinations also may be employed if the fluid reacts in a manner similar to the decomposition of hydrogen peroxide.

### BSPR:

Decomposition of hydrogen peroxide for use in a hybrid motor has been achieved in other prior devices. The most commonly used device is the silver screen catalytic bed. Such a method is patented by Moore et al., disclosed in U.S. Pat. No. 2,791,883. Another prior device uses liquid injection of an aqueous chemical catalytic solution impinging onto a liquid injected stream of hydrogen peroxide. Further, a publication by Pugibet et al. (NASA Technical Translation TTF-13034, 1970.) describes a method using a chemical catalyst as a coating on the surface of a hybrid fuel grain. Finally, the patents of Klein and

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## Document Number 11

Entry 11 of 16

File: USPT

Oct 13, 1981

DOCUMENT-IDENTIFIER: US 4294633 A

TITLE: Blasting composition

## ABPL:

A blasting composition of high solids content, usually of plastic solid consistency, is composed of 60 to 90 parts by weight of solid particulate oxidizer salt, which is mainly ammonium nitrate, preferably fertilizer grade prills, and 10 to 40% of liquid slurry partially filling the interstices and voids in and between the solid particles. The slurry is one that will not appreciably dissolve or soften the granules, hence it is preferably a substantially saturated and thickened solution, in non-aqueous solvent, of strong oxidizer salt, typically ammonium and/or calcium nitrate to which sodium nitrate may be added. The preferred solvent is a low molecular weight polar organic liquid, selected from the group consisting of methanol, ethylene glycol, formamide and ethanol. In some cases isopropanol or propylene glycol may be included. The slurry is of greater density than the bulk granular solids so that it will significantly increase the overall bulk density of the composition which should contain enough dispersed air to improve its sensitivity to detonation over that of a similar composition containing no air filled voids.

## BSPR:

Another disadvantage of slurries is that at their natural densities they usually will not detonate reliably unless they are sensitized, e.g. aerated by inclusion of finely dispersed gas, either as minute bubbles or enclosed within fine hollow particles such as styrene beads, microspheres of glass filled with air, or the like. Gas bubbles are often generated in situ in the gel, as by decomposing a gas-generated material such as hydrogen peroxide or a nitrite or carbonate salt in the slurry, often requiring close pH control, along with a catalyst to promote gas formation. Although this is effective in some cases, where the explosive is loaded into deep boreholes, with a high hydrostatic load of explosive above, the bottom part of the charge often is so much compressed that the aeration is ineffective and the bottom part of the charge may fail to detonate, even though part of the charge above shoots satisfactorily. In such cases, much more gasing agent or aeration, or the equivalent, must be used at the bottom than at the top and this causes difficulties. The slurry tends to rise out of the water filled hole as it is poured or pumped into place. Highly trained personnel are often required to manage slurry satisfactorily, e.g. by pulling the filling hose up at a carefully controlled rate as the charge is pumped into place, aside from the care required in preparing the explosive mixture. Changes in formulation, from bottom to top of the hole are often required and this requires skilled manipulations. Packaged slurry products often have densities around 1.1 g./cc. and high detonation velocities.

BSPR:

According to the present invention, a filler material is also used but it is one which can be better controlled as to sensitivity. It contributes substantial energy and does not adversely affect the solid particles of ammonium nitrate which make up the main bulk of the charge. It is based on the discovery that certain non-aqueous types of slurry can partially fill the interstices and pores in and between the granules without softening or partly dissolving the granules themselves. This maintains good consistency as well as good sensitivity of the combined solid granules and slurry. The slurries used for this purpose, according to my invention, are based on organic liquid solutions of ammonium and/or calcium nitrate, with or without some other salts, wherein the solvent is a polar liquid, a low molecular weight organic liquid compatible with water and having reasonably good solvency for the nitrates, along with other powerful oxidizer salts, selected from the group which consists of methanol, ethylene glycol, formamide and ethanol. In some cases isopropanol and/or propylene glycol can be used as part of the solvent. As much salt as is practical should be dissolved in the solvent for maximum benefit.

BSPR:

The invention may be described in summary as being a plastic or pourable but predominantly solid composition of strong blasting power, consisting of 60 to 90 parts by weight, based on the total, of particulate oxidizer salt of which at least a major part is ammonium nitrate. The remainder may be other solid particulate oxidizer and/or particulate fuel or a liquid fuel such as oil fuel which coats the particles of oxidizer without significantly dissolving them. The interstices of the forementioned major component are partially filled with 10 to 40% by weight of a liquid, preferably viscous gelled liquid solution wherein the solvent is an organic liquid of relatively low molecular liquid with strong oxidizer salt dissolved in it. The particulate salt comprises the major part of the composition. The liquid portion is essentially inert towards the particulate salts, that is, it does not appreciably dissolve them. It is selected from those organic solvents which have reasonably good solvency for ammonium and/or calcium nitrate, though less solvency than water, selected from the group which consists of the lower molecular weight aliphatic alcohols, the lower aliphatic diols and the lower aliphatic organic amines. Specifically, the solvents are preferably selected from the group which consists of methanol, ethylene glycol, formamide and ethanol, in that order. In some cases, propanol, especially isopropanol may be used or propylene glycol, and any two or more of these may be mixed together and used as solvent for the strong oxidizer. The latter may contain minor proportions of sodium nitrate and/or one of the sensitive and powerful chlorates or perchlorates which are conventionally used in explosive compositions of this type.

DEPR:

Reference will first be made to the single FIGURE of drawing which is attached hereto. It shows the relative solvency of water and various low molecular weight organic liquids for ammonium nitrate. Solubilities of other nitrates are analogous. For reasons of economy, the prilled ammonium nitrate, as prepared for use as agricultural fertilizer, is a preferred solid oxidizer. It will be noted that water is the most effective solvent, in terms of weight nitrate salt dissolved over a reasonable and convenient range of temperatures. Next to water, formamide, ethylene glycol, methanol and ethanol follow in that order. The solvency of ethanol for nitrates is somewhat marginal; for these reasons the other organic solvents listed are usually preferred but ethanol can be used satisfactorily in many cases. All of these organic solvents have a flatter temperature gradient than water and at temperatures well below the freezing point of water, some of them may actually have higher

solvency than water. They are thus advantageous for use at low temperatures, as is known in the prior art mentioned above and they have been used as antifreeze agents in some cases in the past. For purposes of the present invention, however, they are preferred over water because solutions based on them can be used to partially fill the interstices and pores in solid granules or prills of AN and the like without dissolving or softening AN.

CCOR:

149/2

CCXR:

149/43

CCXR:

149/46

CCXR:

149/60

CCXR:

149/61

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Entry 4 of 6

File: USPT

Oct 15, 1996

DOCUMENT-IDENTIFIER: US 5565150 A

TITLE: Energetic materials processing technique

**DEPR:**

Suitable solvents are preferably selected from commonly used organic solvents such as ketones, esters, and alcohols. Typical ketones include acetone and methyl ethyl ketone (MEK). Typical esters include acetates such as methyl acetate, ethyl acetate, and butyl acetate. Typical alcohols include methanol, ethanol, isopropyl alcohol, and propanol.

**DEPR:**

Suitable solvents are preferably selected from commonly used organic solvents such as ketones, esters, and alcohols. Typical ketones include acetone and methyl ethyl ketone (MEK). Typical esters include acetates such as methyl acetate, ethyl acetate, and butyl acetate. Typical alcohols include methanol, ethanol, isopropyl alcohol, and propanol. Acetone is a currently preferred solvent. If necessary, a mixed solvent system may be used.

**DEPR:**

Typical oxidizing agents include high performance solid nitramines commonly used in explosive compositions, such as RDX, HMX, CL-20, ADN (ammonium dinitramide), TNAZ (1,3,3-trinitroazetidine), and mixtures thereof. The high explosive composition typically contains from 80% to 90% oxidizer, by weight.

**DEPR:**

Suitable solvents are preferably selected from commonly used organic solvents such as ketones, esters, and alcohols. Typical ketones include acetone and methyl ethyl ketone (MEK). Typical esters include acetates such as methyl acetate, ethyl acetate, and butyl acetate. Typical alcohols include methanol, ethanol, isopropyl alcohol, and propanol.

**CLPR:**

27. A method of processing a high explosive composition as defined in claim 26, wherein the oxidizer is selected from RDX (1,3,5-trinitro-1,3,5-triazacyclohexane), HMX (1,3,5,7-tetranitro-1,3,5,7-tetraazacyclooctane), CL-20 (2,4,6,8,10,12-hexanitro-2,4,6,8,10,12-hexaazatetracyclo[5.5.0.0.sup.5,9 0.sup.3,11]-dodecane), dodecane), ADN (ammonium dinitramide), TNAZ (1,3,3-trinitroazetidine), and mixtures thereof.

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Entry 17 of 28

File: USPT

Apr 18, 1989

DOCUMENT-IDENTIFIER: US 4822433 A

TITLE: Emulsion explosive composition

**BSPR:**

The oxygen-supplying component of the discontinuous phase suitably comprises any oxidiser salt capable of releasing oxygen in an explosive environment in an amount and at a rate sufficient to confer acceptable explosive characteristics on the emulsion composition. Inorganic oxidiser salts conventionally employed in the production of emulsion explosive compositions, and suitable for inclusion in the compositions of the present invention, are disclosed, for example, in U.S. Pat. No. 3,447,978 and include ammonium salts and salts of the alkali- and alkaline-earth metals--such as the nitrate, chlorate and perchlorate salts, and mixtures thereof. Other suitable salts include hydrazine nitrate and urea perchlorate. The oxygen-supplying component may also comprise an acid, such as nitric acid.

**BSPR:**

Ammonium nitrate is preferably employed as a primary oxidiser salt comprising at least 50% by weight of the oxygen-supplying salt component, supplemented, if desired, by a minor (not exceeding 50% by weight) amount of a secondary oxidiser component, such as calcium nitrate or sodium nitrate. A secondary oxidiser component may be incorporated into an aqueous discontinuous phase but its presence is particularly desirable if the oxygen-supplying component is to be incorporated into the emulsion in the form of a melt, i.e. in the substantial or complete absence of water from the discontinuous phase. Suitable secondary oxidiser components which form an eutectic melt when heated together with ammonium nitrate include inorganic oxidiser salts of the kind hereinbefore described, such as the nitrates of lead, silver, sodium and calcium, and organic compounds, such as mono- and poly-hydroxylic compounds including methanol, ethylene glycol, glycerol, mannitol, sorbitol and pentaerythritol, carbohydrates, such as glucose, sucrose, fructose and maltose, aliphatic carboxylic acids and their derivatives, such as formic acid and formamide, and organo-nitrogen compounds, such as urea, methylamine nitrate and hexamethylene tetramine, and mixtures thereof.

**BSPR:**

If desired, the emulsion composition may additionally comprise a solid oxidiser component, such as solid ammonium nitrate or ammonium perchlorate--conveniently in the form of prills or powder, respectively.

**DEPR:**

A mixture of ammonium nitrate (76.7 parts), and water (15.5 parts) was heated with stirring to a temperature of 85.degree. C. to give an aqueous solution. The hot aqueous solution was added, with rapid

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Entry 1 of 3

File: USPT

Oct 26, 1999

DOCUMENT-IDENTIFIER: US 5972136 A

TITLE: Liquid propellant

**ABPL:**

A liquid oxidizer for propellants or explosives comprising nitric acid (Hsub.3), ammonium nitrate (NH.sub.4 NO.sub.3), and water.

**BSPR:**

The most effective liquid oxidizer presently used in the propellant compositions is inhibited, red, fuming, nitric acid (IRFNA), which produces excellent energy and performance as a component of the propellant compositions. However, IRFNA is very toxic and extremely corrosive, which increases the cost of the systems hardware in which it is used; IRFNA's corrosiveness has resulted in system leakage causing damage to the weapons or other systems and personnel injuries.

**BSPR:**

A further object of this invention is to provide energetic liquid explosive and propellant oxidizers that are less corrosive and easier to handle than the concentrated hydrogen peroxide and fuming red or white nitric acids presently in use.

**BSPR:**

These and other objects of this invention are achieved by providing solutions comprising nitric acid, ammonium nitrate, and water.

**BSPR:**

The liquid oxidizers of this invention are solutions of nitric acid (HNO.sub.3), ammonium nitrate (NH.sub.4 NO.sub.3), and water. They are referred to as ANNA (ammonium nitrate-nitric acid) oxidizers. The oxidizers form two classes: (1) concentrated (or high energy) ANNA oxidizers and (2) dilute (or lower energy) ANNA oxidizers. The concentrated ANNA oxidizers are useful as oxidizers for liquid gun propellants or liquid explosives. The dilute ANNA oxidizers may be used as oxidizers for liquid propellants for air bags, torpedoes, and aircraft carrier catapults systems.

**BSPR:**

The concentrated ANNA oxidizers comprise nitric acid (HNO.sub.3), ammonium nitrate, and water in the following proportions. The HNO.sub.3 comprises preferably from about 30 to about 95 and more preferably from 50 to 81 weight percent of the concentrated liquid oxidizer. Nitric acid or HNO.sub.3 here refers to the pure compound (100%) HNO.sub.3. The ammonium nitrate comprises preferably from 5 to about 40 and more preferably from 10 to 30 weight percent of the concentrated liquid oxidizer. The water comprises preferably from about 8 to about 30 and more preferably from 8 to 20 weight percent of the concentrated liquid oxidizer. The water may be sea water, but preferably it will be fresh water, and still more preferably it will

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## Document Number 28

Entry 28 of 28

File: USPT

Mar 19, 1974

DOCUMENT-IDENTIFIER: US 3797392 A

TITLE: REVERSIBLE SENSITIZATION OF LIQUID EXPLOSIVES

## BSPR:

In a similar manner, it is possible to sensitize any potential detonable liquid, or liquid mixture, and it is possible to similarly initiate a liquid phase which phases into a liquid flushed oxidizer salt, such as, nitromethane and ammonium nitrate, with the sensitizer means in its "overdriving" configuration, followed by nitromethane, unsensitized, followed by nitromethane flushed ammonium nitrate. In this illustration the entirety should detonate.

## BSPR:

Tertiary chemical explosive systems -- Dithekite (any of several mixtures being ratios of Nitric Acid/NitroBenzene/Water i.e., Dithekite 13 is 63/24/13), these can be formed for cap sensitivity and reduced from sensitivity by including a diluent greater amount of nitrobenzene or nitric acid staying inside the miscible phases of the three phase diagram.

## BSPR:

Hydrogen Peroxide/Water/(Organic substances such as ethanol) is a family of explosives providing rates from about 750 to 7,000 m/sec when initiated with a booster and a detonating cap. These explosives were investigated by Shanley and Greenspan and the mixtures can be constructed onto triangular phase diagrams which identify detonation regions.

## DEPR:

Ammonium Nitrate (AN) added to the sensitizers did not enhance the effectiveness, though detonation was achieved in some instances. On the other hand in initiating into AN from the overdriven velocity liquid NM where the AN is flushed with NM initiates very well, detonating still above the velocity usual to NM for the distance considered, in the extension described.

## DEPR:

While there are many liquid explosives and liquid explosive mixtures which can be used with such a reversible means according to the invention, specifically they include: desensitized nitroglycerin and other nitric acid esters, nitroalkanes, including nitromethane and mixtures of nitroalkanes, liquid oxygen (LOX) liquid explosive mixtures, Sprengel type explosive mixtures, EGDN, isopropyl nitrate, Dithekite, hydrazine or hydrazine nitrate containing explosives, etc.

## DEPV:

An = ammonium Nitrate added

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Entry 26 of 28

File: USPT

May 15, 1979

DOCUMENT-IDENTIFIER: US 4154633 A

TITLE: Method for making solid propellant compositions having a soluble oxidizer

**ABPL:**

An oxidizer that is soluble in a polar solvent selected from ethanol, diethyl ether, and acetone is used in a propellant composition mixing procedure to effect an improved dispersion of the oxidizer. The soluble oxidizer is selected from the reaction products formed by reacting together an equivalent amount of a first reactant selected from triethanol amine or diethanol amine with an equivalent amount of a second reactant perchloric acid when the soluble perchlorate salt is desired for use. The soluble oxidizer is selected from the reaction products formed by reacting together an equivalent amount of a first reactant selected from triethanol amine or diethanol amine with an equivalent amount of a second reactant nitric acid when the soluble nitrate salt is desired for use. The dissolved oxidizer is added to the binder portion of the propellant during mixing. After being widely dispersed, the soluble oxidizer and binder portion are mixed under low vacuum and mild heat (e.g., 110.degree. F. to 120.degree. F.) to remove the polar solvent thereby leaving the oxidizer in a widely dispersed state. The other propellant ingredients are added and mixing is continued to achieve homogeneity prior to forming and curing the propellant for its intended use. The propellant composition with the widely dispersed oxidizer shows improved burning rate.

**BSPR:**

The oxidizer is selected from the triethanol or diethanol amine reaction products formed from reacting the specified amines with nitric acid or perchloric acid. The reaction products are prepared by reacting equivalent amounts of the reactants. The reaction products of triethanol amine and perchloric acid are polytriethanol amine perchlorates with an average molecular weight of about 600. Other reaction products are polyethanol amine perchlorates or polyethanol amine nitrates with molecular weights of up to about 600. The reaction products are dissolved in an appropriate amount of a polar solvent which can be easily removed under low vacuum and mild heat as specified. The preferred polar solvent is selected from ethanol, diethyl ether, and acetone. The polar solvent, water, is not preferred for use alone; however, it can be used in combination with the polar solvent ethanol. In actuality, ethanol contains about 5% water. If a combination containing water is used, processing will require more controls. For example, the water in combination with isocyanate curable compositions should be processed in accordance with established procedures to avoid gassing in the finished propellant grain; otherwise, propellant grains with voids and cracks could result.

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Entry 19 of 28

File: USPT

Oct 6, 1987

DOCUMENT-IDENTIFIER: US 4698105 A

TITLE: Water-in-oil type emulsion explosives

**BSPR:**

Since the first disclosure by U.S. Pat. No. 3,161,551, various modified inventions have been achieved for the W/O type emulsion explosives such as in U.S. Pat. Nos. 3,242,019, 3,447,978, 3,715,247, 3,770,522, 4,008,108 and 4,482,403, and G.B. Patent No. 1,593,163. The W/O type emulsion explosives in these inventions basically comprise as the continuous phase (oil component), mineral oils, waxes, other hydrophobic carbonaceous fuels, as the discontinuous phase an aqueous solution of oxidizing agent mainly composed of ammonium nitrate and W/O type emulsifier. By further adding optionally sensitizing agents such as nitric acid, strontium ions or hollow microspheres thereto, sensitivity widely ranging from booster initiation to No. 6 cap initiation can be obtained. It has been well known that these W/O type emulsion explosives exhibit high detonating velocity which can not be attained in conventional slurry explosives and are extremely safe as compared with dynamites.

**BSPR:**

The oxidizing agent used in the present invention includes ammonium nitrate, alkali metal nitrates, alkaline earth metal nitrates, alkali metal chlorates, alkaline earth metal chlorates, alkali metal perchlorates, alkaline earth metal perchlorates and ammonium perchlorate and used alone or in admixture in water.

**BSPR:**

It is possible to use, as an auxiliary solvent, water-soluble organic solvents such as methanol, ethanol, formamide, ethylene glycol and glycerine in the aqueous solution of the oxidizing agent for lowering the crystallization temperature.

**DEPR:**

W/O type emulsion was prepared by melting 0.5 part by weight of liquid paraffin, 2.0 parts by weight of microcrystalline wax (manufactured by Esso Petroleum Co., trade name: Eslux 172), and 1.0 part by weight of paraffin wax (manufactured by Nippon Petroleum Co., trade name : 145.degree. paraffin) at about 90.degree. C., by admixing under stirring an aqueous solution of oxidizing agent comprising 69.8 parts by weight of ammonium nitrate, 10.0 parts by weight of calcium nitrate, and 12.0 parts by weight of water previously heated to dissolve at about 90.degree. C. and 2.7 parts by weight of a mixture comprising sorbide stearic acid ester, sorbitan stearic acid ester, sorbitol stearic acid ester, stearic acid and sodium stearate as the emulsifier in which the mixing ratio is 2.3/39.7/52.0/2.5/3.5 by weight, the ratio of sorbide stearic acid ester/sorbitan stearic acid ester/ sorbitol stearic acid ester is 2.4/42.2/55.4 by weight, and the ratio of monoester/diester/triester is 1.5/1.0/0.5 by weight. W/O type